AMERICAN
DYESTUFF REPORTER

VOLUME XXII JULY 17, 1933 NUMBER 15

The Conditions of Temperature and Relative Humidity in Testing Fastness to Light By Means of Artificial Illumination*

By R. O. HALL
Department of Textile Research, Ontario Research Foundation

The mean conditions of temperature and relative humidity under which materials are exposed to the carbon arc in certain existing fading equipments have been determined. The temperature at the exposed face of the sample is in general higher and the relative humidity lower than the values for the Standard Sun Test, recommended by the American Association of Textile Chemists and Colorists. A new equipment is described which embodies accurate control of both temperature and relative humidity at predetermined values.

Introduction

IN COMPARING the fastness of colored materials to light, it is necessary to expose them in such a way as to produce the same kinds, and the same relative amounts, of fading as the materials would experience in service. The possible methods of testing by exposure to sunlight or daylight have been explored in a comprehensive series of investigations, notably those conducted under the auspices of the American Association of Textile Chemists and Colorists[1]. On the basis of this work the Association has formulated and recommended a Standard Sun Test for which the following merits are claimed:

"It is possible to obtain practically reproducible fading from day to day for five or six months of the year, for we have found that from April to October there is relatively little variation in the sun's fading activity between 9 and 3 on sunny days."[2]

"The results of tests have been shown to be reproducible at different times of the year and in different parts of the United States."[3]

This method of exposure may be expected to produce the same type and the same relative amount of fading as will be experienced in service by a great variety of materials used for different purposes. It is, however, subject to recognized limitations. In the first place, its use is restricted to the six months of the year, April to September inclusive, and continuous investigation of fastness problems throughout the year is therefore impossible. Secondly, the method is comparatively slow, since it can only be used between 9:00 a.m. and 3:00 p.m., and on days when the sun is not obscured by cloud. In regions where the summer climate is characterized by a dearth of sunny weather the consequent delay and uncertainty can be a serious hindrance in testing. Hence there is needed a more rapid and convenient method of fading materials than is provided by exposure to sunlight, and this need justifies the endeavor that has been, and is still being made, towards establishing a reliable method of using artificial light twenty-four hours a day at any period of the year.

In view of the dependability of the Standard Sun Test it is desirable that all the conditions of exposure in this test that have any influence on fading should be exactly reproducible in any method of testing by exposure to artificial light, or, since exact reproduction is impracticable, the nearest possible equivalent should be sought. Amongst the principal factors that influence the fading of dyed textiles two of the most important are temperature and

*Read in part before the Washington meeting of the American Chemical Society, March, 1933.

relative humidity, and the values that are significant in this respect are the values for the air in close contact with the exposed faces of the materials. The constant conditions of temperature and relative humidity that are approximately equivalent to the fluctuating values in the Standard Sun Test have been determined for Toronto and found to be as follows:[4]

Temperature of the air surrounding the material	19°C.	66°F.
Temperature at the face of the material during exposure (mean values for a dark plush and a white worsted flat cloth)	44°C.	111°F.
Mean temperature elevation at the face of the material	25°C.	45°F.
Relative humidity of the air surrounding the material	53%	
Calculated relative humidity of air raised to the temperature of the fabric face	12.9%	

In the course of certain fastness standardization work being carried out it was found necessary to determine the conditions of temperature and relative humidity to which fabrics are subjected when exposed in the best fading equipments commercially available, and to compare these condition with the average conditions of the Standard Sun Test.

An Examination of Fading Equipments Using Artificial Light

(1) *Description of the Instruments Used.*

It has been established qualitatively[5] that the relative fading power of energy radiated at different wave-lengths is dependent to some extent on the position and nature of the absorption bands characteristic of the material. For this reason it is essential in artificial tests to use a source of light whose energy distribution resembles that of sunlight as closely as possible. The data available[6] concerning radiation from various lamps indicate that the carbon arcs are the most satisfactory of all sources giving sufficiently intense illumination to be of practical utility. Two instruments that have been developed commercially for testing fastness to light, and which embody carbon arcs are the Fade-Ometer and Fugitometer. For testing fastness to light of dyed materials both these instruments have proved of great value, not only in commercial testing but also in investigational work on fading. The Fade-Ometer, which is manufactured in the United States of America, has been modified from time to time, improvements being made with a view to more accurate duplication of the atmospheric conditions of sunlight fading. The Fugitometer, manufactured in Great Britain, was developed in collaboration with the British Research Association for the Woollen & Worsted Industries, and in this instrument, as in the Fade-Ometer, attention has been given to artificial humidification of the air surrounding the test samples. Experiments by Cunliffe, in his researches on fading, have shown that the humidity within the exposure cells is too low and modifications using separate water bubblers before each cell have been employed to raise the relative humidity to 60-65% (Cunliffe—J. Soc. Dyers & Col. 1931, *47*, 7). This method is stated by Cunliffe, however, not to be completely satisfactory. In the present paper the results of experiments given relating to the Fugitometer have been obtained on the type of instrument which has been marketed commercially and is described below. In the Fade-Ometer and Fugitometer examined the lamps were of essentially the same design, both consisting of enclosed flaming arcs between plain electrodes, and operating on direct current of the same power.

The Fade-Ometer used was of the type CV, in which, by means of a fan built into the top of the instrument, air is drawn upwards past the lamp globe and the samples. Owing to constructional details some of the air passing across the face of the material is drawn directly from the room, while the remainder is drawn through a loosely constructed cotton fabric whose edges are immersed in water. New wicks were used for the present investigation to ensure using the instrument under satisfactory conditions.

In the Fugitometer, no forced air current is provided, as in the Fade-Ometer, to cool the exterior of the lamp. The patterns are enclosed in sample boxes having windows of Vita glass 2 mms. thick. There is no interchange between the air of the laboratory and that surrounding the patterns, for the latter air, after passing through the sample boxes is drawn over the surface of water contained in a chamber below the lamp, and then returned to the sample boxes. For the relative humidity determinations and most of the temperature measurements in this instrument, one of the sample boxes was replaced by another of modified design specially constructed for the purpose. This box had a window in the back through which to make observations, and although having overall dimensions roughly equal to those of the original box, it was made rectangular in all three sections and devoid of internal projections in order to accommodate more simply the required apparatus. The air temperature in this box was found to be identical with that in the normal box.

In all the experiments conducted precautions were taken to avoid interfering in any way with the normal working of the instruments as they would operate in the testing of dyed materials.

(2) *Conditions of Temperature.*

As regards temperature, two values are of interest; that obtaining at the illuminated face of the material and that of the bulk air surrounding it. Both of these temperatures have been determined.

The temperature existing at the face of the exposed material depends to an appreciable extent on the nature of the material itself. Absorption of radiant energy by the material causes the temperature at the exposed face to be higher than that of the surrounding air, and the difference is naturally greatest for the most strongly absorbing materials, such as black materials with a nap or pile. In the present comparison of the different instruments with each other and with the sunlight test, the criterion

used is the mean temperature for a dark plush and a white, worsted flat cloth. This mean temperature was calculated for each instrument and for the sunlight test from measurements obtained on plush combined with data correlating the values for the two extreme types of material. The procedure adopted was as follows. The temperature differences between the exposed faces and the surrounding air were determined independently for the plush and the flat cloths by means of thermocouples[7]. The ratio,

$$\frac{\text{Temp. elevation for white flat cloth,}}{\text{Temp. elevation of dark plush}}$$

was constant over the range considered (10°-35°C.) and equal to 0.65. The mean of the temperature elevations for the two materials was therefore equal to the temperature elevation for the plush multiplied by the factor $(1+0.65)/2=0.83$. The sum of this temperature elevation and the temperature of the bulk atmosphere surrounding the material was taken as the required mean temperature at the face of the exposed materials.

The temperatures actually measured in the two instruments were determined by means of small mercury thermometers. In the case of the bulk air surrounding the material but not enmeshed with it the thermometers were freely suspended and shielded from direct radiation from the lamps. In order to measure the surface temperature of the exposed material the thermometer was stitched to a sample of dark mohair plush of which the pile was just long enough to let the thermometer sink half way into it while leaving the other half exposed. The exposed portion of the thermometer bulb was blackened with a thin coating of soot. It was difficult to be certain from theoretical considerations that this ensemble would register the temperature of the air enmeshed with the exposed face of the material. It was found by direct experiment, however, that the temperatures so obtained were equal to those recorded by means of thermocouples, and that the correspondence was independent of the intensity of illumination over the range considered. The agreement between the figures obtained by the two different methods is illustrated in Table I. The thermo-electric measurements were obtained by means of five chromel-alumel junctions stitched to the face of the plush at intervals of ¼ inch apart, and consisting of spheres about 2 mms. in diameter.

Table I

Temperatures recorded via thermo-junctions (°C.)	*Corresponding temperatures recorded by the mercury thermometer (°C.)*
36.9	37.6
46.4	47.1
56.1	56.6
62.3	62.0

In graph (1), Fig. 1, are shown the temperature observations recorded for the plush material at intervals of two minutes in the Fade-Ometer and in graph (2) Fig. 1, the corresponding temperatures for material exposed in the Fugitometer. The large fluctuations of temperature observed in both instruments are due to rotation of the flame about the axis of the electrodes. This causes the positive crater, from which most of the radiation is derived, to face opposite sides of the instrument in turn. The ultimate mean temperatures obtained for the Fade-Ometer (type CV) and the Fugitometer, and the values determined for the Standard Sun Test when used in Toronto are given in Table II to the nearest whole degree.

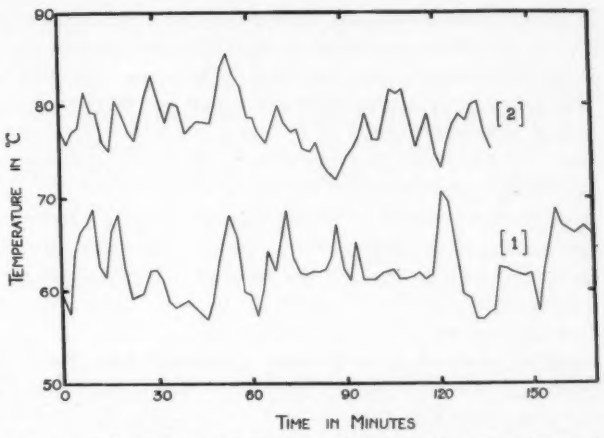

Fig. 1—The Variation of Temperature at the Face of the Material Exposed to the Violet Arc. [1] in the Fade-Ometer; [2] in the Fugitometer.

Table II

Equipment	*Temp. of air surrounding material* °C.	°F.	*Temp. at the face of the material* °C.	°F.	*Difference* °C.	°F.	*Laboratory Temperature* °C.	°F.
Fade-Ometer (type CV)	28	82	62	144	34	62	25	77
Fugitometer	50	122	78	172	28	50	25	77
Sunlight Test	19	66	44	111	25	45	..	..

(3) *The Conditions of Relative Humidity.*

In so far as the rate of fading is concerned, the effective moisture content of a dyed material is that of the exposed face during the fading process. This moisture content is governed by the temperature of the material and the relative humidity of the air in contact with it. Since the fabric is at a pronouncedly higher temperature than the surrounding air it follows that a relative humidity gradient must also exist in the neighborhood of the exposed face. The accurate determination of relative humidity in a small local region where sharp gradients exist would be difficult if at all possible by direct experiment. However, an approximate estimate of the values corresponding to the temperature measurements may be calculated from the temperature data, the relative humidity of the bulk air surrounding the materials and the known saturation pressures of water vapor at different temperatures. The values so obtained are given in Table III. These values are not recorded in the belief that they are necessarily exact estimates of the true mean relative humidities that exist in the several cases, but that they are, like the temperatures recorded above, suitable criteria for comparative purposes.

They are calculated on the assumption, for which evidence exists, that the temperature of the air enmeshed with the irradiated face of a fabric is the same as that of the fibers themselves. It is not to be expected that the values so obtained will be far removed from the truth, and for purposes of comparison, irrespective of their absolute accuracy, they are of value.

The relative humidity of the bulk air flowing past the material in the Fugitometer was obtained by determining the equilibrium weight of a ball of wool (0.2 gms.) whose weight at various humidities and the approximate temperature was known. This part of the investigation was conducted by means of a small quartz-spiral micro-balance[8] enclosed in the special sample box and shielded from the direct radiation from the arc. The value of relative humidity found in this instrument was that attained by the air when using pure water in the humidifying chamber. In the Fade-Ometer the corresponding relative humidity is susceptible to variations that depend on the weather and the difference between the indoor and the outdoor temperatures. Hence, to determine a reliable average value would necessitate investigations extending over a long period of time and under a variety of local conditions. In a limited series of determinations made in the Fade-Ometer type CV a mean value of less than 40% was obtained, while Jameson[9] states that for the improved Fade-Ometer (type FDA) the value ranges from 52% to 60%. The true mean value for the Fade-Ometer type CV probably lies between 40% and 56%. Hence two calculated values of the mean relative humidity at the exposed faces of materials are given in Table III corresponding to the values 40% and 56% for the air flowing past the material.

Table III

Equipment	*Mean relative humidity of the atmosphere surrounding the material*	*Calculated mean relative humidity at the exposed face of the material*
Fade-Ometer (type CV)	(40%	(6.9%
	(56%	(9.6%
Fugitometer	34%	9.6%
Sunlight Test	53%	12.9%

Discussion

From the investigational results given above it is to be seen that in the Fade-Ometer (type CV) and in the Fugitometer the temperature at the exposed face of the material is higher, and the relative humidity lower, than the corresponding average values in the A.A.T.C.C. Standard Sun Test.

In the Fade-Ometer (type CV) no means are provided for regulating temperature or relative humidity at predetermined values, and variations occur according to the changes in the atmospheric conditions of the room in which it is operated. As previously stated, no experiments have yet been conducted on the new Fade-Ometer (type FDA), but it is stated by Jameson[10] to operate at a lower temperature than the type CV and to embody a similar but improved humidifier. A thermo-regulator is incorporated for controlling the temperature of the exposed samples at a fixed value, this regulation being effected by automatic operation of the fan blowing air upwards through the humidifier and past the samples. This control will be effective up to a definite maximum room temperature the value of which will depend on the difference in temperature between the face of the material and the upward current of air. Since the relative humidity of the air surrounding the patterns is not subjected to independent, automatic control it is not to be expected that it will remain constant, but that it will depend to some extent on the atmospheric conditions in the neighborhood of the instrument. The relative humidity will also depend to some extent on the temperature control, since the degree of artificial humidification will be greatest when the cooling fan is in operation and blowing air through the moistened wicks.

By using a fan under the Fugitometer to create a strong upward draught past the lamp, the temperature of the air flowing through the sample boxes, and therefore also the temperature at the face of the material, can be reduced by approximately 15°C. The increase in relative humidity is less pronounced because the temperature of the water in the humidifying chamber is also reduced by the fan. Conditions in the Fugitometer are improved materially by using a fan beneath the lamp, but they still differ from the conditions found for the Standard Sun Test.

For purposes of the present comparison the mean conditions obtaining in the A.A.T.C.C. Standard Sun Test have been used as criteria of the conditions in exposure to sunlight. It can be shown that in vertical tests, and in many service conditions of exposure, the temperature of the material is lower and the relative humidity of the air in contact with it is higher than in the Standard Sun Test at all latitudes outside the Arctic and Antarctic zones. The differences found between artificial and sunlight exposures would therefore be increased if the vertical method of sunlight testing were considered.

In order to duplicate the average conditions found for the Standard Sun Test, and to make possible the accurate control of temperature and relative humidity at different known values for investigational or other purposes, the equipment described below has been constructed.

The Design and Construction of the New Equipment

In the new experimental instrument that has been constructed for fading colored materials the lamp itself is an enclosed carbon arc of the type used in the Fade-Ometer and Fugitometer[11]. It is encircled by a thermostat in the form of an annular tunnel 10 ins. x 12 ins. in cross section, and constructed of sheet brass and copper. The latter contains a thermo-regulator and heating coil, a hair hygrometer with relay contacts, glass windows facing the arc, and sample holders to which access is gained through

sliding hatches in the top of the tunnel. Much of the heat radiated from the arc is prevented from reaching the tunnel by means of a screen placed between the two. This screen has openings opposite the tunnel windows, and the space between it and the tunnel is swept by a current of air from a 12 inch fan situated below the arc lamp.

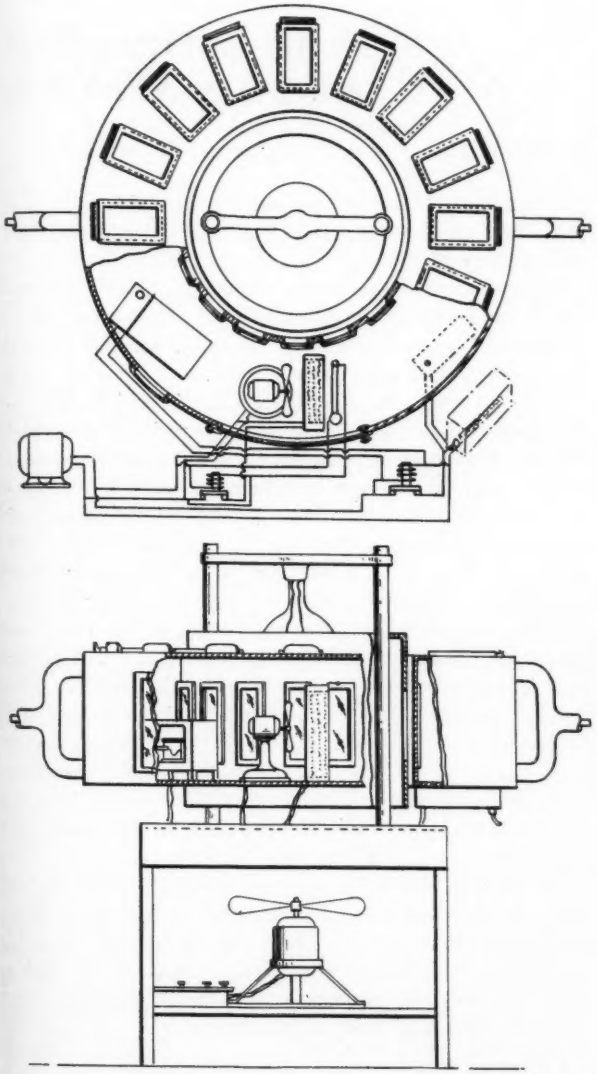

Fig. 2—A New Design of Fading Equipment for Exposure to Artificial Light Under Controlled Conditions of Temperature and Relative Humidity.

The radiant heat that reaches the tunnel and some of the heat conducted to it from the air of the laboratory (when the latter is at a higher temperature) is removed by a stream of cold water flowing through the double outer wall of the tunnel. Scale drawings illustrating the essential features of the new instrument are shown in semi-diagrammatical form in Fig. 2. The upper drawing is a plan in part section showing the disposition of the control equipment, the fan for circulating the air in the tunnel, and the form of the hatches through which access is gained to the samples. The latter are not shown, but are situated parallel to and one inch from the windows. The lower drawing is a side elevation also in part section to illustrate the screen, the windows and double wall of the tunnel, and the fan used beneath the lamp for blowing air upwards.

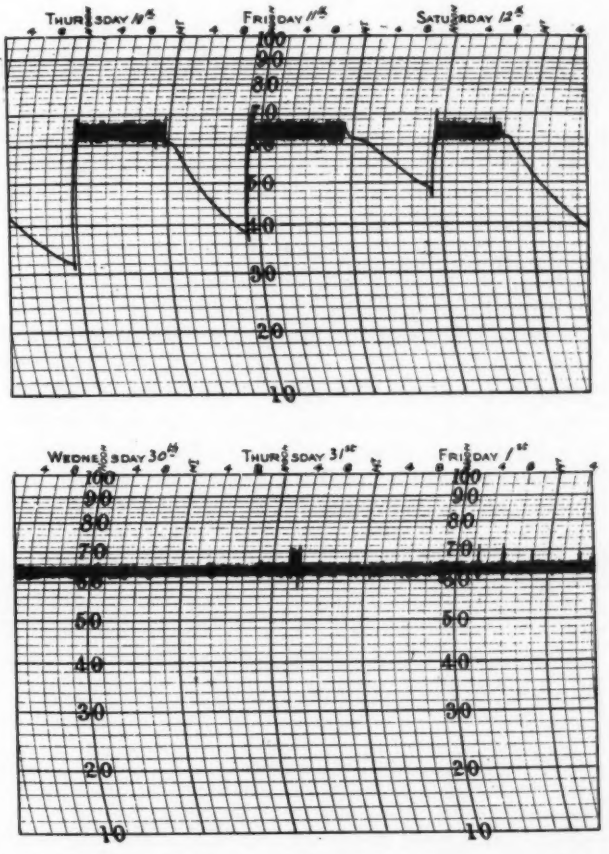

Fig. 3—Automatic Recordings of Relative Humidity in the New Fading Equipment. Top—Intermittent Operation. Bottom—Continuous Operation.

In the first experiments with this instrument humidification was obtained by means of a compact evaporator of small volume in which steam was generated by a knife type immersion heater taking 250 watts. In addition to the time lag of about one minute between completion of the circuit and the commencement of evaporation, this humidifier was a source of unwanted heat. Consequently it was replaced by a centrifugal atomizer of the type used for household and factory humidification, and simpler control of humidity was thereby obtained.

(Continued on page 444)

Tables for the Microchemical Detection And Estimation of the Metals

By KARL M. HERSTEIN, F.A.I.C.
Consulting Chemist

Introductory Remarks

MOST of us have been held back from adopting microchemical methods by two considerations; first, that a large investment in special apparatus was required, and second that a complicated technique would have to be learned for which most of us have not the time. Today, microchemistry has progressed to the point where neither of these considerations is valid. Beyond an ordinary medium power microscope, a few slides and cover glasses, a few small platinum loops, and some glass tubing and an assortment of small size test tubes; nothing is needed but a good selection of reagents for the different elements. The technique, in general, is simpler than that of ordinary quantitative analysis, and the need for much practice and skilled judgment in interpreting results can be avoided by the frequent use of comparison standards of known composition.

On this basis the attached tables have been compiled to assist in the selection of a suitable test for any special purpose without the necessity of studying through the voluminous, and frequently inaccessible literature which microchemistry has developed in the last two decades.

Three types of reactions have been included in the tables. The first type, comprising the great majority of the tests consists in mixing reagent with a solution of the sample and by the development of a characteristic color show the presence of the sought for element, and by the intensity of the color offers a means of quantitative measure of the amount of that element present.

Next, there is the type of reaction in which a drop of the solution to be tested is placed on a suitably prepared test paper and the development of a color in the spot serves to identify the element. While this method of procedure lends itself to the detection of the smallest traces of material, it does not, unfortunately, permit of more than guesses of a quantitative nature.

Finally there is the type of reaction in which the production on a microscope slide of characteristically formed crystals, which can be recognized under a suitable magnification, usually about 100 diameters, serves both for identification, and very often for a measure of the amount of the element present. This type of test is performed by placing a drop of test solution on a *thoroughly clean* microscope slide, placing near it a drop of proper reagent and drawing a platinum wire on fine glass fiber from one drop to the other so that the two flow together. Usually no stirring, heating or other treatment is needed. The preparation is watched through the microscope and the formation of crystals whose size and shape, compared with those formed in a similar manner between the same reagent and a solution of known composition, will give an approximately quantitative measure of the presence and amount of the sought for element.

Throughout the tables the necessity of separating interfering elements has been indicated. In general, these separations, where not specially described are to be performed by the well-known methods of qualitative analysis. Emphasis should always be placed on keeping the solution volume as small as possible.

ELEMENT Reagent	Reaction Type	Separation	Procedure
SILVER p-Dimethylamino-benzal-rhodanine.	Color.	By sol'n in 5% Pot. Cyanide.	Treat sample with Cyanide, filter, add one drop 0.03% sol'n of reagent in acetone and 2 drops 2N HNO_3. 50 times as sensitive as Chloride reaction.
MERCURY Diphenyl-carbazide.	Spot.	As Mercuric Nitrate from other metals.	A neutral sol'n spotted on paper impregnated with a fresh sol'n of reagent in alcohol. Sensitivity from 0.0001 mg. up depending on acidity.

ELEMENT Reagent	Reaction Type	Separation	Procedure
LEAD Diphenyl-thiocarbazone.	Color.	From zinc.	A sol'n containing Pot. Cyanide is shaken wi. a sol'n of reagent in CCl_4. The green colo. changes to brick-red.
COPPER Thiocyanate plus Pyridine.	Color.	By treatment of Chloride or Nitrate sol'n with NH_4OH and filtering.	Add to the neutral sol'n 1 cc of 25% KCNS, 2 drops of freshly distilled Pyr. and shake out wi. a measured vol. of $CHCl_3$ layer. Sensitivity 0.01 mg.
BISMUTH Thiourea.	Color.	With other metals of group II.	Add to a dilute acid sol'n a 20% sol'n of reagent. Sensitivity 10 parts per 1,000,000.
CADMIUM Diphenyl-carbazide.	Spot.	With copper from other metals.	A cold saturated sol'n of reagent in 90% alcohol is sat. wi. KCNS and 2% KI added and poured on filter paper. Spot with sample and fume wi. NH_3. In a few minutes a clear blue violet appears. Sensitivity 0.008 mg.
TIN a-Dinitro-diphenylamine-sulphoxide.	Color.	From reducing agents.	The HCl sol'n is treated with pure Zn. in a current of CO_2. When the Zinc is dissolved add 2cc 0.2% reag. in 0.1N NaOH. Heat 2 min. dilute add 2 drops $FeCl_3$ 10%. Filter. Lauth's Violet is formed. Sensitivity 0.25 mg.
ARSENIC MERCURIC Bromide paper	Color. Gutzeit test.	As $MgNH_4AsO_4 6H_2O$	The p'ptate is dis'd in As free HCl and reduced wi. KI and $SnCl_2$. The cold sol'n is treated wi. Zn and the H_2 passed over special paper. See Methods Off. Agr. Chem. Sensitivity 0.01 mg.
ANTIMONY Pyrogallol.	Micro-crystals.		To one drop of slightly *acid* sol'n of sample add a crystal of pyrogallol. Small angular characteristic crystals in acid sol'n form. Sensitivity not stated.
IRON Thioglycollic Acid.	Color.		To 5cc sol'n add one drop reagent and 0.5 cc. excess strong NH_4OH. Sensitivity 0.002 mg.
CHROMIUM Diphenyl-carbazide.	Color.	From oxidizing agents and Group II metals.	Add sol'n of reagent in one part glacial Acetic Acid nine parts Alcohol to acid sol'n of sample containing Cr. as Chromate. Sensitivity 0.00025 mg.
ALUMINUM Aurin-tri-carboxylic acid.	Color.	From Group I & II and from Fe by sol'n in NaOH.	To 5cc sol'n in dilute HCl add 5cc 20% Amm. Acetate 5cc 0.1% sol'n reagent, let stand 5 min. and make alkaline wi. NH_4OH plus $(NH_4)_2CO_3$. Sensitivity 0.01 mg.
MANGANESE Benzidine	Spot.	From oxidizing agents. Gold, Cerium, etc.	On filter paper place one drop test sol'n, add excess 0.05% NaOH and one drop 0.05% sol'n reagent in 10% Acetic Acid. Sensitivity not stated.
NICKEL Potassium Dithio-oxalate.	Color.	Heavy metals by H_2S. Iron as basic Acetate.	To the filtrate freed as stated add a few grains (5-10 mg.) solid reagent. Compare in 50 cc. Nessler tubes wi. standards from 0.01 to 0.1 mg. Nickel content.
COBALT Nitroso-R. Salt.	Color.	From heavy metals.	To 2 cc. of test solution add 1 g. Sod. Acetate and 2 cc. 0.5% sol'n reagent in water. Boil, add 1 cc. HNO_3 conc. Boil 1 min. compare wi. standards. Sensitivity 0.06 mg.
ZINC Resorcin	Color. (Two-tests are given as neither is perfect.)	From heavy meals wi. H_2S. Iron, etc. wi. NH_4OH.	To 1 cc. alcohol sol'n 10% reagent add one drop ammonia. On standing color finally changes to blue. Sensitivity 0.002 mg.
		USE CONTROL TESTS.	
ZINC Diphenyl-amine.	Color. (These reagents can be used as spot on Fabrics)	As above.	To Acetic Acid sol'n of sample add 5 dr. 1% reagent in glacial Acetic Acid and 5 cc. 5% Pot. Ferricyanide. Sensitivity not stated.
BARIUM Sodium Rhodizonate	Spot.	From all metals of preceding groups.	One drop of test sol'n on filter paper is spotted wi. 1 dr. of 1% reagent in water. A brown-red spot turning bright red wi. 0.1N HCl indicates Ba. Sensitivity 0.00025 mg.
STRONTIUM Sodium Rhodizonate.	Spot.	Same as Barium.	Same as Barium except color disappears in 0.1N HCl. Sensitivity 0.0005 mg.

ELEMENT Reagent	Reaction Type	Separation	Procedure
CALCIUM Picrolonic Acid.	Micro-Crystals.	From Pb., Cu. especially.	Prepare on a *warm* slide wi. test sol'n and cold saturated sol'n of reagent in water or 1% Acetic Acid. Rectangular crystals. Sensitivity 0.0001 mg.
MAGNESIUM Titan Yellow (Thiazol or Clayton Yellow)	Spot and Color.	Other metals especially Sn., Al., Ba., and Ca.	To 10cc test sol'n add 0.2cc. 0.1% reagent in water and make alkaline wi. NaOH. Sensitivity 0.0015 mg.
POTASSIUM 6-chloro-5-nitro-m-toluene sodium sulphonate.	Micro-crystal.	Preceding Groups, and from Ammonium.	Use saturated sol'n of reagent on micro. slide. Warm gently to aid crystal growth. Sensitivity 0.01 mg.
SODIUM Uranyl-Magnesium Acetate.	Micro-crystal and Color.	Not needed.	Add excess (5-10 volumes) of reagent (see note) and stir vigorously. If prepared on a slide the crystals can be identified under the microscope. If prepared in a beaker they can be washed wi. alcohol and weighed or dissolved in warm water and the color measured. Sensitivity depends on volume of test sol'n about 0.1 mg. in 1cc.
(Note:—the reagent is prepared as follows: A. Uranyl Acetate Cryst..... 85 g. Acetic Acid ..... 60 cc. Water to make..... 1 liter. B. Magnes. Acetate Cryst.....500 g. Acetic Acid ..... 60 cc. Water to make..... 1 liter. Mix A. & B. warm to 70°C. cool and filter.)			
AMMONIUM NESSLER sol'n or Chlorplatinic Acid.	Micro-crystal.	Not needed.	Place drop of test sol'n on micro slide. Add drop conc. NaOH, surround with ring cut from a test tube 5-10 mm. high and edges ground. Cover with cover glass having drop of reagent on lower surface. Watch under microscope. Sensitivity 0.001 mg.

TESTING FASTNESS TO LIGHT

(Continued from page 441)

Experimental Tests of the New Equipment

During the winter months, and for considerable periods during the summer, it was found possible to operate this experimental equipment under the required conditions without recourse to refrigeration. The mean values of temperature and relative humidity determined for the Standard Sunlight Test have been maintained to within one or two degrees Fahrenheit and one or two percent relative humidity. In its experimental state as described above, and using tap water not artificially cooled, the thermostat can be operated at about 3°C. below room temperature[12], or at any temperature exceeding this value. During hot weather, and in localities where the supply of tap water is not sufficiently cool, the water circulating through the outer double wall of the tunnel must be artificially cooled by refrigeration.

The mean temperature elevation at the face of the exposed samples can be maintained at any value from 10°C. to 25°C. according to the speed of the circulating fan in the tunnel. The relative humidity of the bulk air in the tunnel can be maintained at any value from about 40% to 90% without the use of drying media, for the excess moisture on humid days condenses on the inner surface of the cold outer wall. No experiments have been conducted at lower relative humidities. The degree of regulation at a mean value of 64% is illustrated in Fig. 3. The upper chart is a record of the relative humidity with intermittent operation over a period of three days while the lower one is a record obtained during continuous operation over a period of three days and nights.

References and Footnotes

[1] Report of the Sub-Committee on Light Fastness: "Light Exposures Series 3," American Dyestuff Reporter, *16*, 707 (Nov. 14, 1927) also "Light Exposure Series 4," American Dyestuff Reporter, *18*, 407 (June 24th, 1929).

[2] Year Book of the American Association of Textile Chemists and Colorists, 1932, p. 116.

[3] American Dyestuff Reporter, *17*, 410 (June 25, 1928).

[4] Full details concerning this work will be available at an early date. Publication is being delayed in order to include data relating to other conditions than those of the A. A. T. C. C. Standard Sun Test.

[5] For a summary of investigations up to 1924, see Cunliffe, Journal of Textile Institute, 1924, *15*, T182.

[6] For a summary see Hadfield, J. Text. Inst., 1927, *18*, T527.

[7] This part of the experimental work was conducted in the determination of conditions in the Standard Sun Test, and will be described in the subsequent publication already mentioned.

[8] J. W. McBain and A. M. Bakr, J. Amer. Chem. Soc., 1926, p. 690.

[9] American Dyestuff Reporter, *21*, 306, 1932.

[10] Loc. cit.

[11] Better lamps may, of course, be substituted when available for the purpose.

[12] The temperature of the water in the outer jacket of the tunnel ranges from a minimum of 4° C. in winter to a maximum of 16° C. in summer.

PROCEEDINGS OF THE
American Association of Textile Chemists and Colorists

JULY 17, 1933

Officers of National Association

President

ROBERT E. ROSE
E. I. Du Pont de Nemours & Co., Wilmington, Del.

Vice-Presidents

WILLIAM H. CADY—ALEX. MORRISON

Treasurer

WILLIAM R. MOORHOUSE
National Aniline & Chemical Co., Boston, Mass.

Secretary

A. NEWTON GRAVES
Franklin Process Co., Providence, R. I.

Councillors

RALPH F. CULVER
H. M. CHASE
CARL Z. DRAVES
WILLIAM D. APPEL
HUGH CHRISTISON
WALTER M. SCOTT

President Emeritus and Chairman of the Research Committee

LOUIS A. OLNEY
Lowell Textile Institute, Lowell, Mass.

Local Sections and Their Officers

Northern New England Section
Leverett N. Putnam, Chairman, Worsted Division, Pacific Mills, Lawrence, Mass.
Clarence L. Nutting, Secretary, Arlington Mills, Lawrence, Mass.

Rhode Island Section
Heyward F. Lawton, Chairman, Borden & Remington Co., Providence, R. I.
C. Brian Wainwright, Secretary, Apponaug Co., Apponaug, R. I.

New York Section
Henry F. Herrmann, Chairman, General Dyestuff Corp., 230 Fifth Ave., New York City.
Thomas R. Harris, Secretary, Herrick & Voigt, 21 West St., New York City.

Philadelphia Section
William A. Stringfellow, Chairman, Ankokas Mills, Mount Holly, N. J.
Charles A. Seibert, Secretary, 39 Chestnut St., Salem, N. J.

Piedmont Section
A. R. Thompson, Jr., Chairman, North Carolina Finishing Co., Salisbury, N. C.
Hugh Puckett, Secretary, Chas. H. Stone, Charlotte, N. C.

South Central Section
Harold Schroeder, Chairman, Dixie Mercerizing Co.
Andrew Kelly, Secretary, Burkart-Schier Chemical Co., Chattanooga, Tenn.

Midwest Section
Carl E. Bick, Chairman, Real Silk Hosiery Mills, Indianapolis, Ind.
Frank J. Acker, Secretary, National Aniline & Chemical Co., Chicago, Ill.

South-Eastern Section
E. A. Feimster, Jr., Chairman, Eagle and Phoenix Mills, Columbus, Ga.
Charles B. Ordway, Secretary, Alabama Polytechnic Institute, Auburn, Ala.

Lowell Textile Institute Junior Section
Joseph J. Pizzuto, Chairman; Raymond L. Matthews, Secretary.

North Carolina State College Junior Section
R. A. Gilliam, Chairman; D. B. Hardin, Secretary.

MEETING, SOUTH CENTRAL SECTION

AN OUTSTANDING two-day meeting of the South Central Section of the American Association of Textile Chemists and Colorists was held in Chattanooga on May 26 and 27, 1933.

On Friday evening, May 26, Dr. Robert E. Rose, national president of the A. A. T. C. C., spoke at the University of Chattanooga on "The Value of Research." The interested public was invited to attend this meeting and an audience of over three hundred responded. Dr. Rose was introduced by Dr. Alex Guerry, president of the University. At noon on Friday Dr. Rose was tendered a luncheon by Harold Schroeder, chairman of the South Central Section. Officers of the Section, and several prominent Chattanoogans were present.

On Saturday evening, May 27, the chemists of Chattanooga and vicinity were invited to meet with the South Central Section at its regular spring meeting. The meeting was presided over by Harold Schroeder, chairman of the South Central Section. Nearly one hundred were present. During the dinner there was a program of music and entertainment.

Dr. J. W. Edwards of the University of Chattanooga spoke, sponsoring a Chemists' Club for Chattanooga chemists of all lines. About thirty-five signified their desire for such an organization, and their intention of joining.

Paper were presented as follows:

"What the Microscope Shows of Textile Fibers." By Sidney M. Edelstein, Dixie Mercerizing Company.

"Microchemical Methods as Applied to Spots on Textile Fibers." By Dr. Irvine W. Grote, University of Chattanooga.

"Some Aspects of Boiler Water Softening." By Harold H. Morrison, Permutit Company.

Mr. Edelstein illustrated his paper with motion pictures and slides. Mr. Morrison also had some very interesting slides.

This meeting was the best attended since the National Meeting in Chattanooga, and did much to heighten interest in the Section. Six new members had been admitted since the last meeting.

Respectfully submitted,

ANDREW KELLY,
Secretary

Proceedings of the American Association of Textile Chemists and Colorists

Microchemical Methods as Applied to Spots on Textile Fibers*

By **IRVINE W. GROTE**
University of Chattanooga

THE examination and analysis of spots upon textile fibers resolves itself essentially into a special application of one or more of the methods of microanalysis.

Micro methods of chemical analysis are a comparatively recent development of analytical chemistry. They were developed largely by the physiological or biochemists, who necessarily work often with very small amounts of material. The material in which they are interested is frequently mixed with large amounts of complex proteins or other substances. An example of this is the necessity of determining accurately the calcium present in blood, where not over 10 mg of calcium are present per 100 cc. of blood, or 1 part in 10,000. Such methods must be not only accurate, but rapid also, since otherwise the patient might die while the chemist is engaged in his analysis. The biochemists have, out of sheer necessity then, worked out many ingenious methods of dealing with small amounts of substances in the presence of large amounts of foreign material. Many of these methods have proved so useful and rapid that they are being adopted largely by other divisions of chemistry.

Four distinct lines of micro methods have been developed. The first of these is "chemical microscopy" or examination and chemical analysis under the miscroscope. This is often called "spot analysis," since the test is most often made in small drops of solution.

The second method of quantitative microanalysis, in which no microscope is used, but rather the usual quantitative analytical methods. These are carried out with delicate balances accurate to 1/1000 of 1/10,000 of a milligram. This allows the use of very small samples.

The third method is colorimetric analysis. This analysis is by development of intense coloration by union of the substance to be determined with dyes or other reagents. This coloration may then be compared with that developed from a known amount of the substance.

The fourth method makes use of the nephelometer, an instrument for detecting and estimating opalescent precipitates. With this instrument, minute traces of suspended precipitates far too small in size and amount to be caught on any ordinary filter, may be estimated from the brightness of the light reflected in comparison with a known suspension of like kind.

The first of these methods, or "spot analysis," is largely a qualitative analytical process, or determination simply of the kind of substance present. The other three are more quantitative in nature, and follow logically the preliminary qualitative method by chemical microscopy. Most often the textile man is interested only in what is causing his trouble, and not how much, although quantitative determinations may occasionally have to be made. We will therefore discuss first the qualitative, and will touch only briefly upon the quantitative methods.

In applying the science of chemical microscopy to the spots encountered on textile fibers, the first operation of course, is to remove enough of such spotting substances from the cloth for examination. As much of the spotted surface as it is possible conveniently to obtain, is cut out of the textile and submitted in small amounts to the action of solvents. At the same time portions of the normal textile fabric are treated likewise. Any foreign substances found present in the damaged portions must be proved to be absent in the normal fabric before they can be blamed as causing the spotting.

If the spot appears greasy, an ether extraction is made with ether known to be free of solids or grease. Such extractions are most conveniently made in 15 cc. centrifuge tubes with cone bottoms. After being stirred about in the tube and then centrifuged, the ether can be separated cleanly and fairly completely by pipetting off the top with a small medicine dropper. After one or two further wash extractions, the extracted fabric may be allowed to dry while examination of the ether extracts is made. These are first evaporated carefully in a micro beaker (which is simply a small lipped test tube), and then applied to a slide.

If oil is present after evaporation of the ether, a distinction between mineral and vegetable oil can be made by applying a drop of caustic solution to the grease to see if saponification takes place. This phenomena is easily observed under a binocular microscope. Care should be taken to run a check with the normal fabric to rule out

* Presented before the South Central Section, May 27th, 1933.

any normal constituents. I might say here that high powered microscopes are not used in such work, magnification of only 10 to 80 being most common. A true wide field binocular microscope is of great advantage. This should be not merely a monocular tube fitted with binocular eyepieces, but with double objectives, so that depth in the image can be observed. If the spot is not fat, but ether soluble, it may be identified by a systematic survey, as laid down in the bibles ot spot analysis. We are fortunate in having two well written texts on such subjects, translated into, or written in English. One is that of Chamont and Mason, of Cornell University, and is called "Handbook of Chemical Microscopy." It is in two volumes, is up to date, easily read by a beginner, and is very thorough. The other is called "Microchemical Laboratory Manual" and is by the Austrian, Friedrich Emich. It has been translated and edited by Frank Schneider, and was published last year. Both texts are published by John Wiley and Sons.

If the material in the spotted fabric is not ether soluble, dilute hydrochloric acid can next be tried, again in the centrifuge tube, and again running a blank with the normal fabric. Similarly, dilute sodium hydroxide extractions can be made. Any dye extracted by such solvents can be removed by taking slightly past the neutralization point and then extracting with ether. If the spot is insoluble in all of these, a digestion may be made with concentrated sulfuric acid in a micro Kjeldahl flask or the material carefully ashed in platinum. Ashing is more dangerous than wet combustion due to danger of volatilizing the small amount of material present. Insoluble substances such as lead sulfate and barium sulfate are easily soluble in H_2SO_4 in the amounts found in microchemical work and can be determined in amounts as small as 0.00003 mg. (0.03 gamma.) Lead is determined by the triple nitrite test, the compound $K_2Pb\ Cu\ (NO_2)_6$ being formed. The term gamma or microgram is used by microchemists to designate 1/1000 milligram.

In making such tests the reagents are best kept in dropping bottles, fitted with capillary end droppers. The reactions are carried out either on a microscope slide or in small capillary tubes held under the microscope. Ultra micro methods can be used for exceedingly small amounts. Such reactions are carried out upon threads previously soaked in the reagent used and then dried, or clean threads may also be soaked in the solution to be tested and then dried. In either case, the treated thread is stretched out under the microscope eyepiece, fixed into place in focus, then a drop of the reagent or test substance is placed on the end of the thread so that the solution will diffuse by capillarity through the thread. In this way, the reaction may be seen to take place slowly, against a white background.

Other methods involving dropping the test solution upon filter paper treated with the reagent, or carrying out the reaction in capillary tubes.

A drop of the solution to be tested can be placed on a slightly greasy slide, and tiny droplets drawn off on a stirring rod from the main drop. These droplets are then mixed with the reagent by drawing a similar droplet of the reagent into it while in focus under the microscope. One drop of the unknown solution may thus be tested with ten or twenty reagents.

A whole system of qualitative analysis has been worked out for such treatments. For instance, a complete qualitative analysis can be made in this way, using a piece of metal alloy weighing not over 1/20 of a milligram, hardly large enough to see with the naked eye. The blood counting chambers used by physicians are very useful for such micro work, as are the measuring pipettes used in blood and urine analysis which have a capacity of 0.05 cc. and measure to 1/1000 cc. Such amounts can actually be filtered, centrifuged, and transferred from one vessel to another without great difficulty, and with the use of fairly inexpensive apparatus.

Specific gravity of tiny droplets of oil may be determined by immersion in an immiscible solvent. A heavier or lighter solvent, dependent upon whether the droplet floats or sinks, is then added until the droplet remains suspended at any level in the liquid. At this point the two are of the same specific gravity. The gravity of the large volume of liquid may then be taken by a spindle or pycnometer, and that of the droplet thus obtained. The refractive index of droplets can similarly be determined by immersing them in an immiscible liquid. There is added then another liquid, miscible with this latter, but of higher or lower index of refraction, until the droplet disappears from view. It then has the same index as the larger volume of liquid, which can of course be measured.

A few of the special reagents used to identify the various metals and anions might be mentioned.

Copper may be detected in amounts of 0.03 to 0.05 micrograms by use of symmetrical diphenyl carbozide or sodium diethyl dithio carbonate.

Nickel may be distinguished by means of dimethyl glyoxine in quantities not exceeding a few micrograms. Aluminum is indicated with aurin tricarboxylic acid; iron with thioglycollic acid, dinitroresorcinol, or kojic acid. Manganese is detected by means of a reaction with tetramethyl diaminodiphenyl methane; cobalt with 3.5 dimethylpyrazole. Magnesium gives a deep blue precipitate with paranitrobenzenediazoresorcinol.

Metals in general can be detected by use of diphenylthiocarbozide.

By consulting the special reagent section of the Organic Chemical catalog of the Eastman Kodak Company you will find a special organic chemical to identify nearly all the separate metals and anions. Many of these are practically specific for an individual metal, even in the pres-

ence of other metals. The old scheme of qualitative analysis might in this way be thrown into the discard, since the student could simply divide his unknown into twenty or thirty test tubes and add the proper reagents. I suspect, however, that even then, most of the reports turned in would be incorrect.

Having determined what substances are present in the spot, a quantitative analysis could then be made if required. Let me warn you again, however, that blank essays should be made with an extract of the normal fabric treated in the same way as the spot material.

Quantitative microanalysis, so called, was developed largely by the work of an Austrian, Pregl, who justly received the Nobel prize for his efforts. Pregl was a biochemist interested in bile pigments and was unable to obtain enough of these substances to submit them to the ordinary organic analytical combustion methods. He therefore decided to drop his biochemical work and endeavor to improve the system of organic analysis, which had been changed but little since the time of Liebig and Wohler a hundred years before. Pregl succeeded in developing a great number of very successful methods of determination, both organic and inorganic. These are well set down in his classic book, "Quantitative Microanalysis," which has been translated by Ernest Fyleman and published by Blakiston. These methods do not differ essentially in theory or method from those ordinarily used, but involve the use of balances weighing to 1/1000 mg. or even 1/10,000 mg. They thus permit the use of smaller apparatus, and 1/100 or less of the amounts of material ordinarily used. Such micro balances cost only $150 to $300. They are very rapid after the technic of weighing has been acquired.

A carbon and hydrogen determination may be made on a 3 mg. to 5 mg. sample; a Dumas nitrogen on 2 mg.; and a Kjeldahl nitrogen on 5 mg. Very exacting technique is required, together with constant practice. After the technique has been acquired, however, accuracy at least equal to the old method may be assured. A great saving in time results also, as micro methods take only 1/4 to 1/5 the time required by macro methods. For this reason, many laboratories have adopted micro methods entirely, even for such things as steel and fertilizer analysis.

Colorimetric analysis is also largely the development of the biochemists. They have worked out quantitative methods for exceedingly small amounts of all sorts of substances, both organic and inorganic. It is really not necessary to point out to a group of dye chemists the small amounts of substances needed to color solutions sufficiently for comparison, when such substances are developed or combined with dyes or other highly colored reagents. The biochemist can take only one drop of blood and determine accurately the sugar content of not over 0.1% of it, with an error of not over 5%. He may have, from the same drop, enough samples left over to determine urea, uric acid, or creatinine. Such methods might well be used by textile chemists in difficulty with spotting. Probably the best reference work on colorimetric analysis is that of Yoe, called "Photometric Chemical Analysis," in two volumes, published by John Wiley & Sons. This monumental work lists some thousands of references on almost every conceivable substance you might wish to determine. In addition, it gives working directions covering a great many of the substances most commonly used.

Nephelometric methods, or analysis by comparison of light reflected from colloidal dispersions as compared to known strength solutions of like kind, offer great possibilities. A very fine exposition of nephelometry is given in the second volume of Yoe's work on "Photometric Chemical Analysis." This method of analysis may be improved further in the future by comparing micro photographs of such solutions. By such ultra super micro methods we may be able to determine the brand of tobacco used by the workman who insists on spitting in the dye bath.

SOUTH CENTRAL SECTION
Committees—1933

Sectional Committee:

W. S. McNab, Quaker City Chemical Co., Chairman.
Nestor Grotelueschen, Magnet Mills, Clinton, Tenn.
W. T. Sledge, Miller Smith Hosiery Mills, Chattanooga, Tenn.
H. A. Rodgers, National Aniline and Chemical Co.

Member National Nominating Committee:

Robert S. Wheeler, Crystal Springs Bleachery, Chickamauga, Georgia.

A Letter to You . . .

CONVENTION HEADQUARTERS
AATCC
CHICAGO

Dear Member: July 17, 1933.

The Annual Convention will be held in Chicago on September 8 and 9. Your committees have been making preparations for several months. You can best show your appreciation of their efforts by making your plans now to attend.

May we count on you?

THE COMMITTEES

American Association of Textile Chemists and Colorists

Proceedings of the American Association of Textile Chemists and Colorists

Modern Housing At the World's Fair

WOULD you like to have a house of your own, adapted to your living requirements, on a lot of your choosing, for, say, $5,000.00? And would you like to have it completed within ten days of the giving of the order for it? Most people would answer "Yes" to such questions but would qualify it with the condition that it be a house that would satisfy them in every particular.

Those of you who come to the convention in Chicago will certainly go to the Fair, and those of you who go to the Fair should visit the eleven exhibit homes grouped together near the lake front. This exhibit may represent the chief contribution that the Fair brings to the standard of living of modern Americans. Some believe that the revolution in methods of house building will date from June, 1933.

The idea back of it all is that in every necessity of daily life, with the exception of housing, we have profited by invention, experimentation, and merchandising. An automobile that was $5,000.00 in 1920 costs $1,295.00 now. The value and efficiency have increased and the price has gone down.

This exhibit of eleven modern homes shows what new materials, new methods of construction, and new design can do to make the home more attractive, more efficient, and less costly. These homes are designed for living, indoors and out, they seek to cut the cost of building and give more value. You will see when you visit them how attractive they are and since each one of them has figured the cost of construction you will be amazed to learn that the highest cost is only $8,000.00.

There is the Brick Manufacturers' House built virtually all in one piece, held together as a unit by steel rods run through the masonry. The Armco and Ferro Enamel House is unique in that it is frameless, no structural steel being used. It has seven rooms, bath and lavatory, garage, solarium and porch, and the cost, exclusive of equipment is $4,500.00.

Another all-steel, frameless house, with nothing made at the site except the concrete piers, is the pre-fabricated house of General Houses, Inc. They are prepared to erect it for you in the Chicago area for $4,500.00 and will soon be able to erect it in units making possible an almost endless variety of designs, in other sections of the country.

Very attractive is the all steel frame house called the Good Housekeeping House. The Rostone House is built of Rostone, a building material composed of limestone and shale and can be had in any color. It has six rooms and costs $6,000.00.

The "Design for Living" home is the creation of John Moore, New York architect, and gives much space to outdoor living and recreation.

The Masonite House takes full advantage of new materials and the walls of one of the bedrooms are covered with broad loom woven cellophane with hangings of knitted cellophane. The Lumber Industries House is built entirely of wood and the walls and ceilings are paneled with various woods, achieving unique designs and demonstrating logical lumber uses.

The "House of Tomorrow" is in the nature of a trial balloon for it is frankly a "laboratory" house for the purpose of determining the attitude of World's Fair visitors to the idea of an utterly different home. The house is built around a central mast which contains all utilities. The exterior walls are of clear glass and there are no windows. Privacy is obtained by Venetian blinds. The ventilation is all by filtered, washed, heated or cooled air, recirculated every ten minutes.

The W. & J. Sloane House displays elaborate interior decoration and shows how well traditional furniture looks in the modern home. All of the houses are furnished but this one is perhaps more in the customary manner of a fine house.

The Glass Block Building draws many visitors as does the Cypress Lodge.

Thus are gathered in one place actual homes using the new materials and new methods of construction which may revolutionize the house building industry. It is one of the best examples of this century of progress and will interest every member who comes to Chicago in September.

See the World's Fair and Attend Your Convention

Proceedings of the American Association of Textile Chemists and Colorists

UNEMPLOYMENT REGISTER

Members of this association, who are without employment, should forward to the secretary a statement as to their qualifications and experience. This information will subsequently appear on these pages with an identifying key number. Employers are also requested to file with the secretary any vacancies which may occur in their businesses.—A. Newton Graves, Secretary, care Franklin Process Company, Providence, R. I.

Key: A—Laboratory and research; B—Dyeing or printing; C—Finishing; D—Sales; E—Instruction; F—Executive

A-1

Education—Ph.B., Brown Univ. '27, P.G. at N. C. State (M.S. this June).

Experience—several years experience as an analytical and research chemist; private research work on sulfonated oils. Twenty-seven years old, married. Will supply references.

A-2

Education—B.T.C., Lowell Textile Institute, 1932; also course in shorthand and typing.

Experience—None to date, interested in quantitative analysis, experimental dyeing, color matching, dye testing and dye identification. Ability to learn quickly and will go anywhere. 23 years old, single.

A-3

Education—Graduate Pratt Institute, also attended Brooklyn Polytechnic.

Experience—12 years laboratory work with dyestuff company, also chemist with chemical company conducting manufacturing and research on sulfonated oils, soaps and specialties. Practical and laboratory experience in the manufacture of textile and leather specialties and starches. Married, 29 years old.

A-B-1

Experience—over twenty years' experience in the laboratories of various chemical manufacturers, four months' experience as second hand in dye house, familiar with all types of dyes and fibers. Thirty-nine years old. Will supply references.

A-B-2

Education—Lowell Evening Textile School, 1917.

Experience—Five years' experience in hosiery dyeing, full fashioned hosiery of silk and cotton and silk, also men's hosiery containing rayon, wool, cotton and celanese. Several years experience in laboratory work. Has also had experience in jig-dyeing of cotton piece goods, slub and top dyeing.

A-B-3

Education—Graduate in chemistry and dyeing of Textile School, Krefeld, Germany.

Experience—2 years plant chemist, handling vat, diazo, and direct fast colors on rayon; 3 years as dyer on piece goods, crepes and all mixtures of celanese, rayon and silk, pure dye and weighted. Age 27, single, will go anywhere. References.

A-B-E-1

Education—B.S. in chemistry at C.C.N.Y. Working now on M.A. at Columbia.

Experience—7 years as dye tester and color matcher for dye manufacturer; 6 months skein silk and rayon dyer; 4 years textile chemist and dyer for cotton finishing concern; 2 months boss bleacher and 4 years as teacher of organic chemistry and dyeing. Age 38, single.

A-E-1

Education—Ph.D., Univ. of Illinois, studied chemical engineering at Columbia, licensed chemical engineer, New Jersey.

Experience—10 years' experience teaching in colleges, subjects included chemical and textile subjects. Has had broad experience in industrial work including experience in testing materials, research and consulting. Will go anywhere; references.

A-E-2

Education—B.T.C., Lowell Textile Institute, 1933.

Experience—Assistant instructor in chemistry, Lowell Textile Institute. Member of scholastic honorary fraternity.

B-1

Education—educated and trained in chemistry and calico printing in Glasgow, Scotland.

Experience—Has been with large print works as colorist for 13 years producing first class work on cotton, rayon, wool and silk fabrics. References.

B-2

Experience—Boss dyer with one firm for 18 years, divisional supt. at print works for 1 year, in charge of processing and dyeing of cottons and rayons at large bleachery for 6 years. Prefers bleachery in East but will go anywhere. References.

B-3

Experience—Foreman piece dyer for the past 21 years. Can dye the following; celanese, rayon, cotton backs, wool, silk and mixed fabrics. Has knowledge of finishing and laboratory work.

B-4

Experience—Overseer of cotton and rayon piece dyeing. Familiar with all machines and all classes of colors. Has knowledge of chemistry, was at one time demonstrator of chemistry and dyeing at Manchester (Eng.) Institute of Technology. Prefers N. E., N. Y., N. J., or Penn., but will go anywhere. Fifty-two years of age.

B-5

Experience—Nine years of yarn dyeing on rayon, silk, cotton, and novelty yarns with acid, direct, vat and naphthol colors. Experience in yarn printing, fastness tests and also on dyeing silk piece goods and rayon knit goods. Will go anywhere; references.

B-6

Education—Graduate R. I. School of Design.

Experience—5 years in bleachery, 7½ years as assistant dyer, 2 years as boss dyer and 3 years as superintendent of dyeing and finishing. Married, 33 years old. References.

D-1

Education—Graduate textile course, 1911, Georgia School of Tech.

Experience—Has had experience in all branches of the textile industry but chiefly in sales work. Was at one time in the dyestuff business and later in hosiery manufacturing. Prefers Middle Atlantic or southern states.

F-1

Education—Textile Institute, Alexander Hamilton Inst., Extension work at Brown Univ., working knowledge of German.

Experience—Has had executive departmental work with all types of textiles manufacturers. Acquainted with all types of textile machinery, dyestuff application, laboratory practice, cost methods, and checking methods and processes. References.

B-D-F-1

Experience—Has held positions as general superintendent of a print and dye works and a finishing company; held a position as boss dyer in a bleachery for 6½ years and as a colorist on 11 printing machines for 3½ years; has held a position as boss dyer for 6½ years with a finishing company and received general training for 3½ years; and has also been dyer in a mill dyehouse. Desires position as superintendent, colorist, dyer or salesman and solicitor.

Accommodations for Members in Chicago

Although the official headquarters for the A. A. T. C. & C. Convention in Chicago on September 8th and 9th will be at the Congress Hotel, the Committee has obtained the names of the following bureaus to whom inquiry may be made concerning other living accommodations, particularly those members who are going to Chicago with their families and might desire light housekeeping apartments rather than regular hotel accommodations.

Travel Guild, Inc., 180 N. Michigan Ave., Chicago, Illinois. Telephone Franklin 4080.

Visitors & Tourists Bureau, 213 S. Wabash Ave., Chicago, Illinois. Telephone Harrison 9445.

Other information may also be obtained from a renting guide which the Chicago *Herald & Examiner* publishes every week. It is also understood that Chicago Chapters of leading lodges and orders of the country have a list of quarters available for members of these bodies.

If persons are desirous of obtaining further advance information concerning living quarters in Chicago for the Convention, it is suggested that they write to:

A. J. Feit,
c/o E. I. du Pont de Nemours & Co.,
7 S. Dearborn Street, Chicago, Illinois.

AMERICAN
DYESTUFF
REPORTER

LOUIS A. OLNEY, Sc.D.
Professor of Chemistry and Dyeing, Lowell Textile Institute
Directing Editor

NORMAN A. JOHNSON
Managing Editor

MYRON DREW REESER
Advertising Manager

Published biweekly by
HOWES PUBLISHING CO., INC.
440 Fourth Avenue, New York City

A. P. HOWES, President
M. D. REESER, Secretary
H. F. CRAWFORD, Treasurer

Subscription price: $5.00 per year; Canadian $6.80; foreign $6.00

Vol. XXII July 17, 1933 No. 15

CURRENT COMMENT

TODAY is rather a significant date in textile manufacturing history in the United States, inasmuch as it marks the official day for the code of the textile manufacturers, as framed by the Cotton Textile Institute, to become effective. The textile industry, one of the largest in the country, has done well by itself in having its code of fair competition to be the first approved by the President under the provisions of the National Recovery Act.

The idea of government control of industry has never been exceptionally popular and private industry would probably rather work its way out of the depression in its own manner. However, it is the belief of the administration that it is only with government supervision that the speediest exit will be effected. Sir Josiah Stamp, British financier and economist, after expressing his approval of President Roosevelt's policy in an international radio broadcast, went on to say:

"Although I do not think that the control of industry can ever have more than a limited success at best and may create great insecurity at its worst, people on this side of the Atlantic do not realize the depths of the difficulties into which the United States' price level collapse and banking collapse have brought her industrial activities and how necessary it was for such striking measures to be put forward, first for the psychological reactions that were necessary to command confidence, and secondly, actually use them for raising prices if the psychological reaction to their existence was inadequate."

Theoretically the objects and provisions of the Recovery Act are appealing to many and we hope their practical application will earn commendation.

* * *

Among other industries there is still much confusion and delay although the lumber industry has submitted a code which will probably be changed inasmuch as it provides for a 40 to 48-hour week and in the words of Administrator Johnson, "A forty-eight-hour week is so long I wouldn't even consider it." Further this code proposes a wage scale ranging from 22½ cents per hour to 45 cents per hour. The lower figure does not meet with the approval of Mr. Johnson, as it is far below what he considers as a necessary minimum wage. Public hearings for this bill are set for July 20th and many of the existing recommendations will in all probability be changed to meet the approval of the administration.

* * *

Among other happenings of the past two weeks was the formation of an advisory council by the President to speed progress by the administrations recovery machinery. This council will consist of all Cabinet members and the administrators of the special federal agencies recently set up by Congress. It was regarded by the President that such a council was necessary in order to get all of the recent legislation into effect within the next month. Speed seems to be the essential requirement in order to get the provisions of the Recovery Act into practical operation.

In order to put more men to work Secretary Ickes has obtained the approval of the Cabinet Advisory Board of the Public Works Administration for a list of Federal projects totaling $200,000,000. Those works have been selected which will put the greatest number of men to work in the shortest possible time.

* * *

And now we hear of exorbitant price rises. It has always been natural to have prices go up before wages have been raised, but the policy of the administration has been expressed as being decidedly against any profiteering activities. We elaborated upon this subject to some extent in our last issue. The complaints so far have not been leveled at our industry but against increases in bread prices. In regard to this matter, Secretary Wallace recently telegraphed the mayors of 49 large cities in 25 states warning that complaints against profiteering were being turned over to Attorney General Cummings for investigation. To prevent unwarranted distress wages must keep pace with prices, inasmuch as such action is possible and profiteering must be stopped at its source.

* * *

Another group that is beginning to make itself heard now is that faction that was clamoring for the government to do something during the latter part of the Hoover administration when the President could not obtain the power that is vested in the present incumbent, and now that recovery indications are becoming prevalent, are just as strong for no government supervision and urge that industry be allowed to take its natural course. What's the answer?

NEW PATENTS

(*Abstracted by S. O. C. M. A.*)

Vat Dyestuffs. (Obtained by condensing dibenzanthrones with polynuclear heterocyclic compounds; dye cotton dark blue, greenish blue or grey to black shades of excellent fastness.) Max Albert Kunz, of Mannheim, and Karl Koeberle, of Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,909,691, May 16, 1933.

Production of New Vat Dyestuffs of the Anthraquinone Series. (New and extremely fast vat dyestuffs giving grey to black dyeings on cotton in a single operation, obtained by treating the products produced according to application Ser. No. 267,478, filed April 4, 1928, with alkaline or acid condensing agents, in the presence if desired, of solvents or diluents, such as trichlorbenzene, aniline and like.) Max Albert Kunz, of Mannheim, and Karl Koeberle, of Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,909,692, May 16, 1933.

Vat Dyestuffs of the Dibenzanthrone Series. (Comprises condensing a pyranthrone with a dibenzanthrone in the presence of an inert organic diluent of high boiling point; dye vegetable fiber grey to black shades of excellent fastness; a continuation in part of copending application Ser. No. 267,478, filed April 4, 1928.) Max Albert Kunz, of Mannheim, and Karl Koeberle, of Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,909,693, May 16, 1933.

Vat Dyestuffs of the Dibenzanthrone Series. (Comprises condensing a vat dyestuff containing a perylene ring system and a plurality of substituents selected from the group consisting of halogen and the nitro group with a dibenzanthrone in the presence of an inert organic diluent of high boiling point; dye cotton blue to black shades of excellent fastness. A continuation in part of copending application Ser. No. 267,478, filed April 4, 1928.) Max Albert Kunz, of Mannheim, and Karl Koeberle, of Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,909,694, May 16, 1933.

Vat Dyestuffs of the Dibenzanthrone Series. (Comprises condensing a polynuclear aromatic compound containing a plurality of substituents selected from the group consisting of halogen and the nitro group but which itself does not possess vat dyestuff properties, with a dibenzanthrone in the presence of an inert organic diluent of high boiling point; dye cotton dark blue to black shades of excellent fastness. A continuation in part of copending application Ser. No. 267,478, filed April 4, 1928.) Max Albert Kunz, of Mannheim, and Karl Koeberle, of Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,909,695, May 16, 1933.

Intermediate for Azo Dyes. (Relates to new derivatives of 2-hydroxy-3-naphthoic acid, free from carboxylic or sulfonic acid groups.) Emmet F. Hitch, of Wilmington, Del., assignor to E. I. duPont deNemours & Co., of Wilmington, Del., No. 1,909,960, May 23, 1933.

Sulphur Dye and Process of Making the Same. (Comprises fusing an aromatic nitro compound with an alkali metal polysulphide in the presence of a sulphite-cellulose compound; yield brown dyes of excellent shade and fastness.) Herbert A. Lubs, of Wilmington, Del., and Paul C. Bowers, of Penns Grove, N. J., assignor to E. I. duPont deNemours & Co., of Wilmington, Del., No. 1,910,441, May 23, 1933.

Polymethine Dyestuff. (Dyeing tanned cotton reddish-blue to greenish-blue shades of goods fastness.) Karl Schmidt, of Leverkusen-on-the-Rhine, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y. No. 1,910,478, May 23, 1933.

Polymethine Dyestuff. (Dyeing tanned cotton violet to reddish-blue shades of good fastness.) Karl Schmidt, of Leverkusen-on-the-Rhine, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,910,479, May 23, 1933.

Dibenzanthrone Dyestuff and Process of Preparing Same. (Comprises oxidizing dibenzanthrone compounds by treating said compounds suspended in sulfuric acid with manganese dioxide; dyes cotton from an alkaline hydrosulfite vat blue shades changing to brilliant green on treatment with suitable oxidizing agents.) Robert J. Goodrich, of So. Milwaukee, and John Charles Kinahan, of Milwaukee, Wis., assignors, by mesne assignments, to E. I. duPont deNemours & Co., No. 1,910,603, May 23, 1933.

Azo Dyestuffs and Process of Preparing the Same. (Dye animal fibers, particularly wool, red to yellow to brown bright shades of good fastness to fulling and to light; prepared by diazotizing amino-benzyl-ortho-benzoic acids and coupling to an azo component, e.g., 2-phenyl-amino-5-naphthol-7-sulfonic acid.) Ivan Gubelmann and Joseph B. Oesch, of So. Milwaukee, Wis., assignors, by mesne assignments, to E. I. duPont deNemours & Co., No. 1,910,690, May 23, 1933.

Purification of Vat Dyes and Intermediates. (E.g., a nitrated dibenzanthrone compound, by subjecting it to the action of a mild alkaline oxidizing agent.) Herbert J. West, of Crafton, Pa., assignor to the Selden Co., of Pittsburgh, Pa., No. 1,910,819, May 23, 1933.

Rayon Dyeing and Finishing-10

(Continued from page 434, July 3rd issue)

By **B. L. HATHORNE**
Consulting Textile Chemist

CHAPTER VI—DYEING AND FINISHING OF TRICOT AND MILANESE FABRICS

All Rights Reserved by Author

Discussion of Greige Goods

TRICOT and milanese fabrics containing rayon are commonly produced from the following fibers and fiber combinations:—

(1) Regenerated Cellulose and Real Silk.
(2) Cellulose Acetate and Real Silk.
(3) Regenerated Cellulose and Cotton.
(4) Regenerated Cellulose.
(5) Cellulose Acetate.

The rayon in rayon-containing tricot and milanese fabrics comes to the dye house containing the same types of impurities and added materials as does the rayon in circular knitted rayon fabrics and rayon-containing hosiery. These impurities and added materials are thoroughly discussed in Chapters V and VII dealing with circular knitted fabrics and hosiery.

Fabrics manufactured from two beams are always subject to warp streaks due to the possibility of mixing warp threads during the drawing in operation or during the knitting process (broken threads improperly drawn in).

Warp streaks due to knitting errors augment to a considerable extent the difficulties of dyeing two fiber tricot and milanese combination cloths, as they make it necessary that both fibers used in such cloths be dyed to an almost perfect match.

It is customary to "tack" tricot and milanese fabrics prior to the dyeing operation to protect the face of the fabric during the wet processing operation. The edges of the fabric are loosely sewn together in such a way that the face is completely protected from all mechanical abrasion during the scouring and dyeing operations.

Dyeing and Finishing Equipment

Tricot and milanese fabrics containing real silk are usually degummed in large boil-off tanks which closely resemble skein dyeing tubs. The tanks are usually built approximately three feet wide by two and a half feet deep. The length varies from ten to forty feet depending on the plant and quantity of work processed. They are usually built entirely of wood and are fitted with water pipes, steam, and drainage facilities.

Bleaching tubs resemble boil-off tubs with this exception—care is taken that the inner surface of the tubs contain no iron. Steam pipes, used within bleaching tubs for heating purposes, are made from lead and are covered with wooden slats in order that they may not come into direct contact with the fabric.

The usual type of oval reel dye kettle is used for handling tricot and milanese fabric—extra care being taken that the machines are exceptionally free running. In some instances both the front and back rolls of the machines are power driven.

Tricot and milanese dye houses must, of course, be equipped with the usual type of rotary extractors.

Although tricot and milanese fabrics are customarily scrooped, when necessary, in the dye kettle, it is customary to maintain a special scrooping tub in the finishing room in order to facilitate the application of a second scrooping treatment when necessary. Such "scrooping tubs" are usually built large enough to allow a single cut of fabric to be completely immersed in the scrooping liquor. As these tubs contain the scrooping acids they are customarily lined with acid-resistant metal.

As a matter of fact, scrooping methods that must be employed in order to safely process rayon-containing fabric, are such that a scrooping tub described above should not be used.

Tricot and milanese fabrics are dried under tension on stationary frames known as tenters. These frames consist of large rectangles, the inner edge of which is completely covered with a row of pins by which the fabric is held during the drying operation. A series of steam pipes placed approximately one foot below the fab-

ric supply the heat necessary to drive the moisture from the fabric.

In addition to the stationary drying frames some plants have installed automatic moving tenter frames similar to those used for ordinary woven fabric.

After the drying operation, tricot fabric is sometimes calendered between two large calender rolls that run under extra heavy pressure.

Discussion of Scouring

The quality standards demanded by producers of tricot and milanese fabrics are such that these fabrics must be processed throughout the entire dyeing and finishing operations with especial care. In many ways this is an advantage, as it allows dyers and finishers to use materials and methods best adapted for these fabrics, rather than the cheapest possible process capable of giving results barely commercial as in the case throughout the circular knit goods industry.

The rayon lubricants applied to tricot and milanese yarns before the knitting operation do not constitute a scouring problem as they do in the case of circular knitted goods and hosiery, inasmuch as the quantities of soap, etc., customarily used in connection with these fabrics are sufficiently great to easily and completely emulsify the rayon oil.

Water is a problem to dyers and finishers of tricot and milanese fabric only insofar as it necessitates the addition of extra soap to scouring baths. Lime and magnesium soap spots are rarely ever encountered in tricot and milanese fabrics, first because many of the plants handling these fabrics are located in soft water regions; second because those plants that are located in hard water regions are allowed to use sufficient soap to eliminate this danger.

The presence of gum silk in silk and rayon glove silk fabrics does not constitute a scouring problem, as these fabrics are handled throughout the scouring and bleaching (when necessary) operations in the same manner as are pure silk fabrics.

In the case of gum silk and acetate fabrics it is necessary to run the boil-off bath as mildly as possible to avoid saponification of the acetate.

Glove silk fabrics consisting of all-rayon or rayon and cotton fibers are handled in the same manner as are circular knitted rayon fabrics. As such fabrics have been thoroughly discussed in Chapter V, they will not be discussed again in detail here.

Inasmuch as each mill has its own particular scouring problems, no one procedure can be considered as being entirely typical. The procedures given below were successfully used by reputable mills during the 1930 season:

Scouring Procedure

Regenerated Cellulose Rayon and Real Silk Fabric

(1) Tack fabric—face protected.
(2) Attach to boil-off sticks.
(3) Prepare scouring bath containing 1% to 3% (based on liquor volume) neutral soap.
(4) Raise boil-off bath to the boil.
(5) Immerse fabric.
(6) Move goods by means of sticks every half hour for three hours.
(7) Remove fabric, load into dyeing machine, rinse and proceed with dyeing operation.

Scouring Procedure

Cellulose Acetate Fabric

Cellulose acetate and glove silk fabrics require such slight amounts of scouring treatment that they can be successfully scoured by a mild (20 to 30 minutes) scouring treatment in the dyeing machine prior to the dyeing operation. Neutral soap only need be used as the cleansing agent.

Discussion of Bleaching Problem

Regenerated cellulose rayon and rayon and cotton glove silk fabrics are customarily bleached, when necessary, with sodium hypochlorite as described in Chapter V (Dyeing and finishing of circular knitted rayon fabric). Obviously real silk containing glove silk fabrics must be bleached with peroxide of hydrogen.

(*To be continued*)

Caled Products Company, Inc.

Warren K. Cooley has severed his connection as research director and principal of the National Association Institute of Dyers and Cleaners to become president of the Caled Products Company, Inc. This company distributes various solvents and soaps.

1933 Fall Season Color Card

The regular edition of the 1933 Fall Season Silk Card, just issued by the Textile Color Card Association to its members, is presented in an entirely new form.

The 60 shades are shown in good size swatches of piece dye silk, the same material as used in the confidential advance issue released to members several weeks ago. These double swatches are loose at one end, an arrangement especially convenient for matching purposes.

Outstanding basic tones are portrayed on the first three pages of the card, each accompanied by a lighter complementary shade, offering new suggestions for smart color harmonies. Among the important key colors thus grouped are Mole Taupe and Towngrey, Rubylustre and Vintage Red, Stonebeige and Friar Brown, Blue Iris and Metallic Blue, Frostgreen and Dundee Green.

A special collection captioned "Evenglo Shades" highlights gay brilliant hues for sundown fashions. In this group are Pansy Petal, Tipperary Green, Red Fuchsia, Glory Blue, Tartan Red, Fudge Brown, Tomato Bisque, Palermo Blue, Chamois Beige, Phlox Purple, Arcady and Golden Orange.

Mercerization of Rayon Fabrics

By **ERICH SCHUELKE, Ph.D.**

AS WELL known, all types of rayon—to a lesser degree acetate rayon—lose much of their tensile strength in the wet state. They swell considerably in caustic soda solutions and special precautions are required to avoid damage when rayon is subjected to mercerization with caustic soda. Fortunately the damage is not done during the treatment with the strong caustic soda in the mercerization itself, but during the consecutive washing with water, when the lye is diluted to about 20° Tw. equal to about 10% NaOH.

It is important therefore to go through this dangerous concentration either very rapidly or to wash with solutions of salt, etc., which counteract the effect of the caustic soda. Another way to overcome these difficulties is the pretreatment of the rayon with substances, which have a protective colloid action such as, e.g., gelatine.

For years this problem has been dealt with by many inventors with more or less success. Some of the principal inventions may be described in more detail, some of them giving very good results especially on rayon-cotton fabrics, where a good mercerization of the cotton is aimed at without effect on the rayon. A problem for itself is given by the saponifying effect of caustic lyes upon acetate rayon, which gives this fiber an affinity for substantive cotton dyes.

Mercerizing of fabrics is done in two ways for different purposes. If the fabric is subjected to strong tension while under the influence of the mercerizing lye a high luster is obtained, so characteristic for this process. If the fabric is passing the lye without tension a considerable shrinkage occurs and a crepe effect is obtained. The first process is generally applied on cotton fabrics, which contain some rayon as effect threads, while the second process is applied on all rayon fabrics for creping purposes.

In 1923 the Courtaulds, Ltd., in London developed a process, which was patented under British No. 230,187 and in various other countries, by which viscose rayon was first treated with a 3% gelatine solution, dried and mercerized at 65° F. with a caustic soda solution of 50° Tw., which contained 5% of glycerine, for about 2 to 3 minutes. Tension is avoided in order to obtain creping effect. It is then washed with a 5% sulfuric acid until complete neutralization is obtained, rinsed with water and dried with or without tension. In cases where rayon yarn is to be woven into cotton fabrics, later on to be mercerized, the rayon is first treated in the skein with a solution containing 1½% gelatine and 2% aluminum acetate liquor of 8° Tw., dried and woven into the mixed fabric. Then this fabric is mercerized as usual for cotton, washed with acid and finished as required. The aluminum acetate renders the gelatine rather insoluble in water and this gel acts as a protective medium for the viscose rayon. A similar process had been previously described by Heberlein & Co. in Wattwil in the Austrian Patent No. 458, using starch or glue solutions as protective colloid.

W. Marshall of Greenwood Lee in the British Patent No. 210,484 worked out a process for mercerizing cotton fabrics containing acetate rayon threads. The caustic soda in usual strength is applied at a temperature of 15° C. or below, the fabric being passed through under tension. The acetate is partly saponified and thus its affinity for cotton dyes is increased, but its appearance, luster and weight are not impaired. Caustic soda of 22-27% is applied for as short a time as possible at low temperature. The fabric is passed quickly through rinsing baths, cold acid and fresh water. The cotton or acetate rayon may be dyed before mercerizing.

In the British Patent No. 295,488 caustic potash is recommended for mercerizing fabrics containing rayon. E. Ristenpart has described experiments in Melliand's Textilberichte, 1921, p. 130 f, which he made on ordinary cotton in order to determine the mercerizing effect of caustic potash compared with caustic soda. He comes to the conclusion that potash may be used with the same results as soda, provided that it is used at a 50% higher concentration than the molecular weight would indicate. In the British Patent No. 309,280 the Silver Springs Bleaching & Dyeing Co. and A. J. Hall recommend the use of caustic potash of 80° Tw. for the mercerization of fabrics containing acetate rayon. When caustic potash is used the washing can be done with cold water.

In the British Patent No. 295,062 washing with hot water is recommended to follow the application of ordinary coustic soda solution as mercerizing lye. It apparently is important to secure a rapid exchange of the lye in the fiber by water to overcome the swelling effect of diluted caustic soda of a certain critical concentration.

As cellulose hydrate swells more in a cold diluted caustic soda than in a hot solution of the same concentration, hot water is a better rinsing fluid than cold water. The colloidal system involved in this case is comparable somewhat to a soap solution. A perfectly neutral strong solution of sodium oleate congeals at a certain concentration.

If a little caustic soda is added to the system, it becomes more fluid; if the caustic concentration is increased, the soap is "salted out," becomes insoluble and swims on top of the caustic lye. The same effect can be obtained with other electrolytes. Commonly plain salt is used with best effect. How far this analogy reaches is best demonstrated in the British Patent No. 323,305, which recommends hot or cold salt brine for washing the mercerized viscose rayon. It is this method that has found the widest application. Its main drawback though, is the resulting mixture of sodium chloride and sodium hydroxide, which is rather hard to separate economically. More recently a process is finding use by which a 5-10% solution of soda ash is taking the place of the brine in washing. This solution is quite effective and has the great advantage that by a simple treatment with slaked lime all the carbonate is transferred into caustic soda. Perhaps the same effect could be obtained in a still more economical way by passing the fabric, saturated with the strong caustic lye through an atmosphere of carbon dioxide before further washing.

L. Lilienfeld of Vienna claims in his British Patent No. 253,853 that rather diluted caustic soda has a mercerizing effect upon viscose rayon. Concentration below 5% NaOH, preferably below 1%, are used as mercerizing bath, the fabric being under tension. Rinsing is performed with solutions of some salt, ammonium sulfate or common salt, or with 10% sulfuric acid, followed by water.

Other processes were designed to protect the viscose rayon in mixed cotton fabrics by addition of organic solvents to the sodium hydroxide solution. Sam Mansfield Jones in U. S. A. Patent No. 1,316,958 applies the following mercerized bath: to 943 lbs. of 27% caustic soda are added 57 lbs. of denatured alcohol. Less alcohol does not give sufficient protection, while more alcohol reduces the mercerizing effect upon the cotton.

The Arnold Print Works have taken out a series of U. S. A. patents, covering such processes. The alcohol in the above process is replaced in U. S. A. 1,343,138 by the same amount of formaldehyde, which is said to have the same effect. U. S. A. 1,343,139 suggests a mixture of 50 parts of phenol and 950 parts of 27% caustic soda. But not only the monovalent alcohols and aldehydes, but glycerine as well may be used, as their U. S. A. 1,346,802 recommends a mixture of 135 parts of glycerine and 865 parts of 29% caustic soda. This is approaching the method used in the above-mentioned old Courtauld patent, which too adds glycerine to the caustic soda. The esters of glycerine are covered in Arnold's U. S. A. 1,346,803, where a mixture of 181 parts of monoacetine with 819 parts of 32% caustic soda is suggested. In a latter patent, U. S. A. 1,392,833, they replace these organic substances by salts of alkali metals or magnesia or ammonia with preferably organic acids including some inorganic acids, such as nitric and nitrous as well as arsenites, etc. Of these compounds potassium acetate is said to give the best results.

The addition to alcohols and phenols and their derivatives is recommended for ordinary mercerizing in order to increase the penetration of the lye. The I. G. Farbenindustrie has taken out a series of patents, covering the use of sulfonic acid esters, etc., in mercerizing lye, of which especially the German Patents Nos. 446,996 and 545,790 may be mentioned here.

The same concern makes the interesting proposition in its French Patent No. 726,293 to use liquid anhydrous ammonia as mercerization medium on cotton as well as on rayon. Rayon is claimed to be improved in elasticity and affinity for dyestuffs. The mercerizing effect of liquid ammonia is considerably inferior to that of caustic soda, but the fiber is less affected and the removal of the ammonia from the fiber is simple. As liquid ammonia is only stable under pressure the machinery would be quite complicated, which renders the general application of this interesting invention rather doubtful.

Summarily it may be stated, that the mercerization of cotton goods, containing rayon at present can be performed without great difficulty. Of all the processes developed to solve this problem, mercerization with ordinary caustic soda lye, followed by washing with common salt brine or with carbonate solution has found widest application and is most economical.

Fabrics containing acetate rayon cannot be mercerized without at least a slight saponification of the cellulose acetate taking place, which would destroy the cross-dyeing effect so welcome on such fabrics.

National Dyes

Bulletins have been distributed by the National Aniline & Chemical Co. on the following products:

National Sulfur Orange R: an addition to the line of sulfur dyes said to produce much redder and somewhat brighter tones than National Sulfur Orange G Conc. It is said to possess the general all around fastness of the dyes of this class, including excellent fastness to washing, cross-dyeing and sea water, and good fastness to boiling soap and soda, perspiration and fulling.

National Alizarine Sapphire BLN: an addition to the line of acid blues which produces greener and brighter shades than National Alizarine Sapphire BN, but which is practically identical with it in all other respects.

National Erie Fast Orange WS: a new addition to the line of direct dyes. While it produces about the same tones as National Erie Fast Orange A, its superior solubility is said to make it a more desirable product for use in machines, especially those of the package type. The shades produced by this dye are said to possess excellent fastness to light, mineral acids and sea water.

Technical Notes From Foreign Sources

Pure Nitrosamine Printing Pastes

German Patent No. 465,564 (6/IX/'28)—*I. G. Farben-Ind. A.-G.*—The nitrosamine printing pastes as previously prepared have not been pure, or, perhaps better expressed, clean; they contain excess of one or the other components, as the nitrosamine alkali salts, or the passive component, naphthols, hydroxynaphthoic anilides, hydroxy-aryl ketones, acylamino-naphthols, etc., or substances of various sorts which get accidentally into the paste during its manufacture; of which sand or particles of dirt are most dreaded by the printer, as being the cause of streaks, through mechanical interference with the edge of the doctor-blade.

The present procedure covers the purification of the paste by dissolving it, mixed or as the as yet unmixed components, in some suitable volatile solvent, such as acetone; filtering, and then removing the solvent by distillation, at ordinary pressure or in vacuo. Such a product, as paste or dry powder, is of course completely soluble in water, and needs no straining.

Disazo Dyestuffs for Wool (Anthrone Series)

German Patent No. 463,378 (12/VII/'28)—*I. G. Farben-Ind. A.-G.*—The new point covered by this patent consists in the use of a peculiar sort of first component, "dianilo-anthrone," better described, perhaps, as a 1-4-naphthoquinone in which one of the quinone oxygens has been replaced by two aniline radicals, the linkages of the latter to the quinone carbon being in the para position to the amino groups. Such a diamine is tetrazotized as usual, and coupled, e.g., to 1-naphthol-3-6-disulphonic acid, or to pyrazolone-sulphonic acid, two mols. of either. Disazo dyestuffs are thus produced, of course. With the 1-naphthol-3-6-disulphonic acid, a yellowish-red dyestuff is formed, with a pyrazolone-sulphonic acid a yellow, and with naphthylamine sulphonic acids violets, are produced.

Di-o-toludino- and di-o-anisidino-anthrones are also mentioned as first components, giving more bluish products.

Black Tris-azo Dyestuffs

German Patent No. 467,060 (4/X/'28)—*I. G. Farben-Ind. A.-G.*—p-Amino-azobenzene-azo-gamma acid is first produced by diazotizing, e.g., p-amino-acetanilide, and coupling it in acid reaction to gamma acid, then splitting off the acetyl group by hydrolysis after the known methods, diazotizing, coupling to gamma acid in alkaline bath, then diazotizing again, and coupling to m-phenylene diamine. The product dyes cotton and artificial silk (except acetate silk) a pure, fast black. Acetate silk is left untinged by it, which makes possible good dyeings with mixed weaves, with desired color-effects.

It is problematic, however, just what the formula can be. If in its synthesis the acetyl group of the amino-acetanilide be left untouched until the final coupling is over, and only then hydrolyzed, the formula could be deduced easily from our general knowledge of the behavior of such components; otherwise, the product is without doubt, from the chemical standpoint, a bad mixture—which of course does not necessarily alter its value as a dyestuff, unless the possible twofold direction of the reactions produces a mixture of compounds whose percentage varies with each lot. In this latter case, trouble might be encountered in bringing the product to an earlier standard.

Vat Dyestuffs Containing Nitrogen

German Patent No. 468,577 (1/XI/'28)—*I. G. Farben-Ind. A.-G.*—mesmo- Naphtho-dianthrone and its derivatives are of no use as vat dyestuffs, on account of the difficulty in vatting them. But if a negatively substituted meso-naphthodianthrone, e.g., a halogenated derivative, is caused to react with amino-anthraquinones or their derivatives, in which at least one hydrogen is left intact in the amino-group, new and valuable products are formed. The reaction may take place with or without a diluting medium, or with or without catalysts or acid-neutralizing substances.

The halogen derivatives necessary can be obtained from halogen-meso-benz-dianthrones by oxidation or by treatment with anhydrous aluminum chloride, or from meso-naphtho-dianthrone or its derivatives by direct halogenation.

Five examples are given, the products dyeing cotton red-violet, yellow-brown, bordeaux, or dark blue.

Indigoid Dyestuffs

German Patent No. 469,249 (22/XI/'28)—*Gesells. f. chem. Ind. in Basel.*—The halogen substitution products of 2-1-naphtho-oxythiophene are condensed as usual with 2-anils or 2-halogenido-cyclo-o-diketones, the resultant products being in some cases then halogenated. The products (three examples) dye unmordanted cotton from bordeaux to gray.

Indigoid Dyestuffs

German Patent No. 469,342 (22/XI/'29)—*I. G. Farben-Ind. A.-G.*—These products belong to the 2-thionaphthene-2′ indol-indigo series. For this synthesis, 3-oxy-1-thionaphthenes, which must be substituted in the 4, 6,

and 7 positions by either three halogen atoms, one alkyl group and two halogen atoms, or two alkyl groups and one halogen atom, are condensed with alpha derivatives of isatine, or its derivatives or homologues. In the one example given, the product dyes cotton a clear reddish violet from a golden-yellow vat.

Bleaching with Ozone

Wilhelm Bauer—Monats. f. Textil-Ind. 47, 36 (1932). —Perhaps the first idea, from the practical standpoint, in working out methods for the use of ozone in bleaching cotton goods, consisted in the possibility of avoiding, by its use, the more or less harmful action of caustic alkali liquors upon the fiber. The first cautious attempts to employ ozone bleaching involved combination of the hypochlorite and the ozone bleach together; the goods were given a light bleach in the customary hypochlorite liquor, and then exposed to ozonized air for an hour, in closed chambers. In this way, however, the action of the ozone was slight and only superficial; a genuine bleaching through could not be brought about by the ozone in any such way. The real problem to be solved was, however, only a technical one. The principle involved in modern ozone bleaching is simply that of forced circulation of the ozonized air through the goods to be bleached (the hypochlorite treatment is not necessarily combined with it), not simply over the surface of the goods; with provisions for a continuous supply of freshly ozonized air. Machinery is now in use, not only for bleaching of yarns, but also for weaves of full width; the latter machinery can take care of two widths at one time, with a performance of about 300 pieces of 100 meters each, or about 3,000 kilos, in eight hours.

The goods are not tendered, the loss of weight amounts to only about 1%, the goods retain their softness, and the tensile strength is usually improved. As the air used can be ozonized to any slight degree desired, there should exist no danger of over-bleaching. The goods take up the ozone better if previously passed through a weak souring bath, and if the ozonized air is also given a charge of water vapor as it comes into the chamber.

For the bleaching of one kilo of cotton, about 2 g. of ozone are necessary; with the newer ozonizing devices, only about 100-200 watts of current are required. The process is speedy, fewer workmen are needed, operating costs are lower, the goods are more safely bleached. The only factor which keeps the process from rapid adoption seems to be the initial cost of the machinery.

Is Dyeing to Sample Always Possible?

Otto Pennenkamp — Monats. f. Textil-Ind. 47, 38 (1932).—The concluding part of a paper. This subject, if we are not mistaken, has been dealth with before. The present paper deals rather largely with the factors which make exact matching of a sample difficult or impossible.

One point dealt with deserves a thoughtful appreciation. The point is this: if at least the responsible dye manufacturers would simplify their products to a reasonable group of steps, and would also give up the "fancy" names with which too many common dyestuffs are disguised, the problem of the dyer would be greatly simplified. As it is, the dyestuff manufacturers seem to be thinking more of trying to force a given dyer, who uses some of their products, to use none but theirs, since a dyer can hardly be expected to be familiar with all the products of all the manufacturers. Simplification of the range of tones and shades, and of nomenclature, would put the manufacturers upon their mettle, to prove to a dyer that A's Marine Blue BB, for example, is, to be sure, the same dyestuff as their Marine Blue BB, but does not exhaust so well, or does not dye level so easily, etc. To some extent, it seems that the manufacturers are willing to allow difficulty in the dyehouse, if it pushes their sales to a small extent. But a firm whose products are always to be depended upon does not need to worry about increase of sales, nor does a manufacturer who runs his business with purpose to help the consumer, as well as make good profits, suffer by thinking of his trade, at least in the long run. Of course, Ostwald's Color-Atlas is referred to as at present an ideal standard to which to conform.

As the article is really an enumerative discussion of the many factors involved, little can be definitely quoted. The subject is thoroughly taken up.

Acetyl-Cellulose; Past and Present

Monats. f. Textil-Ind. 47, 57 (1932).—A report of an address given by Dr. A. Eichengruen before a meeting of the Verein Deutscher Chemiker.

It was about 30 years ago that the first thin films of cellulose acetate were produced, and the new material was at first looked upon as a possible substitute for the dangerously inflammable cellulose nitrate in its various industrial forms. The cellulose acetate industry which sprang up followed a different line of development, however. The triacetate proved to be not capable of utilization in a technical way, and it was only the discovery of the secondary acetate, by Dr. Eichengruen, about 25 years ago, which made possible the production of acetate silk, safety film, and Cellone, the difficultly combustible celluloid.

It was in 1904 that Dr. Eichengruen began his spinning experiments. It soon proved that the new product, so similar in general to nitro-silk, could not simply take the place of the already established fiber, either in technical use or technical manufacture. As to the latter point, while nitrate silk is spun from below upward, and is already dry at a distance of one meter from the spinneret, acetate silk must be spun in the contrary direction, and the fiber has to be dried artificially as it descends. Since that first discovery, little if anything, certainly nothing essential, has been added to the process. The original samples of the first acetate silk were in all respects the same as the modern product, only the fiber could not then be

manufactured profitably, as no process for recovery of solvent was known. A further difficulty was, that acetate silk was not susceptible to dyeing by the ordinary methods. It was only when these two stumbling-blocks had been removed, that the new fiber became an industrial fact.

Since that time, we have seen acetate silk developed along the lines of delustering, super-lustering, hot-printing of patterns on weaves, etc. The most recent development has been that of Cellone, a difficultly inflammable material of celluloid base. This material has proved to be of the greatest value, not only for such purposes as automobile gogles, gas-masks, etc., but as the base of acetate lacquers, which, in combination with application of really fireproofing materials, have made it now possible to impregnate woven goods so as to make them really fireproof lacquered fabrics. This application is of most value, perhaps, in the production of aeroplane wing fabrics, which as a result of the treatment are not only fireproof, but as elastic as before treatment.

The Blancometer—A Means of Determination of Nearly White Colorings

Monats. f. Textil-Ind. 47, 59 (1932).—The use of the modern photo-electric cell makes its possible to carry out spectrophotometric measurements upon solids or liquids. The field of sight is not involved, the response of the photo-electric cell replacing this standard. The construction of an objective means of measuring color, incorporating the above principle, would be of great usefulness, especially for problems involving the blue end of the spectrum. Some problems which could be solved by the aid of such an instrument would be, the determination of the exact form, and the correlation of the spectral transmission curves of filters, and the sensitivity curve of the photo-electric cells, such as might be in question for definite industrial purposes. The fundamental principles of such an instrument have been described in British Patent No. 324,351.

The new instrument, the Blancometer, is designed for another class of problems, in which mere approximations to the transmission-curves and sensitivity curves are sufficient to afford easily applicable results in the determination and identification of colors, especially in the case of nearly white objects or substances or surfaces. It has already been recognized, that, by use of three suitable chosen filters of considerable transmissive power, the degree of reflection, or the transmissive coefficients, in reference to pure white, when once determined for three corresponding parts of the spectrum, can "locate" a color with a degree of certainty which is extremely useful for the dyeing industry. The eye is often not sufficiently sensitive to detect a color-value which is nevertheless definite in degree, and whose recognition is important. The new instrument has made it possible to extend the recognition of color differences between two almost white samples of material, to a degree about five times as great as before possible.

The principle of the instrument is as follows: A ray of light is reflected (a) from the surface under examination, into a photo-electric cell, after it has passed through two photometric prisms; (b) and then also reflected from a standard white surface of magnesium oxide, after passing through two adjustable prisms. These adjustable prisms make it possible to obtain the same reading in a voltmeter, which translates into a visible numerical value the current generated in the photo-electric cell.

The zero point of the adjustable prisms is determined by observation of two different surfaces of magnesium oxide. When the absorption constant of the prisms is known, then the ratio of the value of the light reflected from the surface under examination, and from the standard white, can be calculated. Light-filters of a definitely known and suitable transmissivity of the spectrum can be interposed, and so make possible a reading off of the red, green, or blue light-intensity as easily as for white.

In order to maintain the illumination (from a Pointolite bulb) at a definite, uniform value, resistances, etc., are attached, with means of varying the voltage to a determined optimum point.

Two diagrammatic cross-sections, and a photograph of the instrument as set up, illustrate the article.

Efficiency of Finishing and Thickening Agents

Franz Koelbl and Dr. L. Zakarais—Monats. f. Textil-Ind. 47, 62 (1932).—Various investigations of the effectiveness of different kinds of starches and gums have already been carried out by means of the Lueers-Herites Gelatinometer. Zakarias has found that the various kinds of starch possess definite degrees of elasticity when their pastes are dried, and also definite thickening-power, which indicates the degree of hardness of the dried paste. The two present authors have re-investigated Z.'s results, modifying his methods somewhat (e.g., adopting a uniform method of producing the paste).

Two tables of numerical results are given, from which the authors derive some definite conclusions:—

a—With potato starch as an example, the figures show that in preparing the paste the starch must be "opened up" in different ways, in order to secure either the greatest thickening power or the maximum elasticity. Up to the present, workers have used different kinds of apparatus, and different kinds of thickening agents, for different purposes, such complications adding to the cost of operation. The present study shows that one can use the same thickening agent for different purposes, by previously determining (with the Gelatinometer) the optimum conditions, and governing the preparation according to his findings.

b—The figures show that, for thickening agents, gels are to be prepared which possess at the highest the same degree of thickening power as of elasticity, that is, substances which do not gelatinize well. It appears, further,

that the efficiency of the paste does not increase in proportion to the concentration, and that the value of some less-esteemed materials, at certain concentrations, are higher than the values of some other more-esteemed materials.

c—The time-factor of rupture of film is not a simple function of the breaking-weight. The graphs are always typical hyperbolæ.

The summary, in 10 paragraphs, partly repeats the above notes. The authors, however, also point out with emphasis, that the Gelatinometer allows the simultaneous determination of elasticity and thickening power of starches, gums, etc.; that the variation of the temperature of paste-formation may, from different materials, produce a paste of the same properties; that the better kinds of materials are characterized by their (definite) thickening power or elasticity lying within narrow limits in reference to each other, but that the elasticity value is usually the higher; that these two properties are reasonably persistent when pastes are stored for a reasonable time, at a fairly high concentration, but that starch pastes deteriorate more rapidly than gum solutions (already known, but now definitely determined as being usual), etc.

Azo Dyestuffs (Dis- or Trisazo-) (Naphthol AS Series)

German Patent No. 427,246 (18/III/'26, issued 20/XI/'28)—*I. G. Farben-Ind. A.-G.*—The Naphthol AS series forms the source of the second component in this group of dyestuffs. The first components are the diazotization products of non-sulfonated amino-azo- or diamino-azo-dyestuffs, which contain at least one free hydroxyl or carboxyl group, or the two simultaneously. Such dyestuffs are diazotized or tetrazotized, and coupled as usual to one or two mols., as the case may require, of a Naphthol AS component, either in substance, or on the fiber, as more usual.

The azo dyestuffs cited as suitable for use as first components may be synthesized, e.g., by diazotization of an appropriate nitro-amine, and coupling it to a phenol, and amino-carbonic acid, a hydroxy-acid, and amino-phenol, etc., followed by cautious reduction of the nitro-group without affecting the azo-linkage; or by diazotization of such a compound as p-amino-acetanilide, coupling it to the above types of second component, and setting free the amnio-group of the acetamino-group by hydrolysis. The diazotization and coupling of these amino-azo dyestuffs to the Naphthol AS components offers nothing new. Two examples are given. The products are claimed to be of excellent fastness, which seems probable, from the structure of the substances.

Acid Azo Dyestuffs Containing Chromium

German Patent No. 463,800 (19/VII/'28)—(*addition to G. P. No.* 411,384 *of* 23/X/'23)—*Gesells. f. chem. Ind. in Basel.*—The original patent is extended to cover the case of other than the originally specified naphthol-sulfonamides or their derivatives, as second components, to be coupled with the diazo-derivatives of o-hydroxy-amines, except picramic acid or o-amino-salicylic acid; and the azo products are treated in the usual way with substances which will introduce copper or chromium into the molecule.

Ten examples are given. The coppered products yield red-violet to blue-violet tones upon wool.

Substantive Disazo Dyestuffs

German Patent No. 469,340 (22/XI/'28)—*I. G. Farben-Ind. A.-G.*—Two earlier German Patents, Nos. 380,-058 and 382,427, cover the production of certain asymmetric dis- and tris-azo dyestuffs, by tetrazotization of 4-4'-diamino-diphenyl-3-3'-dicarbonic acid, and differential coupling with two different second components, one of which must contain sulfonic or carbonic acid groups, or some other group which confers upon the product solubility in water. The present procedure merely limits the coupling on one side to an aryl-naphthylamino-sulfonic acid, the other component being any desired compound which contains the solubilizing groups above-mentioned.

Three examples are given—blue, becoming more greenish by after-coppering, red-violet, and grayish-blue, becoming pure green upon after-coppering. All of the dyeings are fast to light and washing, especially the after-coppered ones, which is only to be expected.

CLASSIFIED

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i.e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.

WANTED, CHEMIST—Experienced in sulfonating oils and manufacturing other textile specialties capable of taking charge of small plant and laboratory in metropolitan New York district. State education, experience, salary, etc., in application. Address Box No. 785, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

Position Wanted: as Boss or Asst. Dyer on Silk Hosiery Full Fashioned and Seamless. Thoroughly experienced in all stages of Hosiery Redyeing and Grey-Stock; close matching; Full particulars and first class references furnished upon request. Salary of secondary consideration; Married, Location no object. Address Box No. 784, American Dyestuff Reporter, 440 4th Avenue, New York City.